Taibah University
College of Pharmacy
Department of Pharmaceutics & Pharmaceutical Technology

PPT 241: Pharmaceutics I

Physical Pharmacy Laboratory Booklet

Prepared by:

Dr. Waleed Neyaz

(wneyaz@taibahu.edu.sa)

2021-2022 1st Semester

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COURSE GENERAL INFORMATION

COURSE TITLE: Pharmaceutics I	**ACADEMIC YEAR: 1443**
COURSE CODE: PPT 241	**TERM: 1st term**
COURSE CREDIT: 3 hrs	**START DATE: 29/8/2021**
PRE-REQUISITE COURSES: **-**	**CLASS TIME:** **Lecture: MONNDAYs** (P3b1 & P3b2 **- 9:00-10:40 am)** (P3b3 **-11:00am-12:00pm** and **1:00-1:40 pm)** **Laboratory: SUNDAYS** (P3b1- **9-11.30 am)** (P3b2- **1-3.30 pm)** **WENSDAYS** (P3b3 **-3.30-6 pm)**
COURSE DELIVERY: In Class Lectures and Laboratory Practical and Tutorial Sessions	**CLASS LOCATION: F35 and F37** **LAB LOCATION: Lecture theater and Pharmaceutics Lab**

COURSE INSTRUCTOR(S)

COURSE CO-ORDINATOR (MALE)	**Name:**	Dr. Mohamed Shaker
	Email:	mamahmoud@taibahu.edu.sa
	Office Location:	Pharmaceutics and Pharmaceutical Technology Department
	Office hours:	________________
	Phone (optional)	Office phone: 3509 Cell Phone:
COURSE INSTRUCTOR	**Name:**	Dr. Waleed Neyaz
	Email:	wneyaz@taibahu.edu.sa
	Office Location:	Vice Deanship of Development and Quality
	Office hours:	Tuesdays and Wednesdays 8:30 am – 10:00 am 3:00 pm – 5:00 pm
	Phone (optional)	Office phone: 014 861 8888 (Ex:3546)
COURSE TA	**Name:**	Mr. Abdellah Al haidri, MSC, BPharm
	Email:	________@taibahu.edu.sa
	Office Location:	Pharmaceutics and Pharmaceutical Technology Department
	Office hours:	
	Phone (optional)	
COURSE TA	**Name:**	Mr. Hatem Al harbi, MSC, BPharm
	Email:	Haharbi@taibahu.edu.sa
	Office Location:	Pharmaceutics and Pharmaceutical Technology Department
	Office hours:	
	Phone (optional)	

ASSESSMENT, EVALUATION AND GRADING

	Assessment task	Week Due	Value (of 100%)
1	**Calculations Assignments (Booklet)**	5	5%
2	**Written weekly reports (Lab Report)**	8-15	5%
3	**Periodical Exam (1)**	7	15%
4	**Periodical Exam (2)**	12	15%
5	**Final Exam (Practical)**	16	20%
6	**Final Exam (Theoretical)**	17-18	40%

Assignment:
Value: 5 % of final grade. Due Date: in the 5th week
Type: [Pharmaceutical Calculation Booklet, weekly assigments]
Description: [The calculation booklet contains Different calculations problems and examples based on the topic covered in the theoretical lecture. Students are required to analyze and solve the examples in the booklet weekly and discus the assignments with course instructors and TAs. Booklet will be evaluated based on the organization, clarity, tidiness, answers and discussion]

Weekly lab reports:
Value: 5% of final grade. Date: weekly in the lab starting from 8th week
Type: [Written Lab report]
Description: [A lab report booklet contains the theory, rational, procedures and required calculations and diagram. At the end of lab session studentds are required to finalize the report and submitted to the Lab TAs for marking. Booklet will be evaluated based on the organization, clarity, tidiness, data, discussion and lab professional behavior]

Periodical Exam (1)
Value: 15 % of final grade Date: TBA Length: 1 hour
Type: In-class
Content: TBA

Periodical Exam (2) Value: 15 % of final grade
Date: TBA Length: 1 hour
Type: In-class
Content: TBA

Final Exam (Practical) Value: 20% of final grade.
Date: in the 16th week Length: 1 hour
Type: Lab work
Content: TBA

Final Exam (Lab) Value: 40% of final grade.
Date: in the 17th and 18th weeks Length: 2 hours
Type: In-class
Content: The entire course content

General Policies and Regulations

ATTENDANCE

- To ensure good academic standing, students are expected to actively participate in all class-related activities. Class attendance and attendance-recording at Taibah University are MANDATORY.
- Students who fail to attend more than 75% of scheduled classes\lab sessions will not allowed to take the final exam
- Attendance will be recorded 10 min after class or lab starts and according to the College\University's policy
- Student who misses a class, lab session, quiz or exam; will need to file a petition to the office of *Vice-Dean for Academic Affairs*. Only students with valid petition will grant a make-up quiz, an examination or a lab test

ACADEMIC INTEGRITY

TU is committed to practice the highest standards of academic integrity and honesty. Students are expected to be aware of University's policies in these aspects. All Students are advised to familiarize themselves with the provisions of the students' rights, responsibilities, and misconducts. Students must avoid any behavior that might possibly result in suspicions of cheating, plagiarism, misrepresentation of facts and/or participation in an offence.

All students must read and be familiar with the Regulations on Students Rights and Responsibilities (https://www.taibahu.edu.sa/Pages/AR/DownloadCenter.aspx?SiteId=77791218-cc53-43c8-bd86-d0540554649a&FileId=49b7470b-0fb6-40b1-b81b-eee3a0af0357)

Academic Misconduct (https://www.taibahu.edu.sa/Pages/AR/DownloadCenter.aspx?SiteId=77791218-cc53-43c8-bd86-d0540554649a&FileId=b8aa57c0-c5bb-4309-98c4-a797c3a9e8bb)

,and other regulations in TU website (https://www.taibahu.edu.sa/Pages/AR/Sector/SectorPage.aspx?ID=110&PageId=8)

LAB POLICIES

- Students are required to adhere to the College's dress code in lab (including scrub, white lab coat and shoes that completely cover the foot). Students who fail to adhere to the dress code will not be allowed to attend laboratory sessions.
- Absolutely No food or drink are allowed in Lab
- Horseplay of any kind is strictly forbidden in the lab.

TRAINING AND PRECEPTOR POLICIES

- Students are required to adhere to the College's dress code in the field-training

- Students are expected to follow the preceptor's regulations.
- Behavioral misconducts are not tolerated in the field-training and can lead to course failure according to the University regulations.

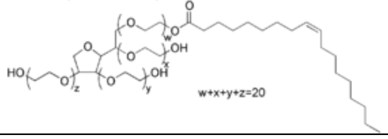
COVID-19 AWARENESS

- If you do not feel well, stay at home: If you have typical COVID-19 symptoms stay home and contact the course instructor and your academic advisor
- Keep your distance: Stay at least 1 meter apart from others and avoid gathering and crowding in the hallways
- Wear a mask all time in classroom and hallways
- Wash/sanitize your hands properly and frequently

GENERAL POLICIES

- Electronic Devices Policy: Laptops and tablets can be used in class (*not in Lab*) for note taking only. Cellphone use is not permitted
- Emergency procedures: Students must familiarize themselves with the nearest Emergency Exit to the Classroom and College Safety and Evacuation Plan
- Class recordings: Not allowed

Lab # 1

Ppt 241: Physical Pharmacy The Determination of the Surface Tension by Drop-Weight Method	
Purpose	To measure the surface tension (γ) of Polysorbate 80 (Tween 80) solution using Stalagmometer  Chemical formula $C_{64}H_{124}O_{26}$ Molar mass 1310 g/mol
Requirements	• Stalagmometer (Figure 1) • 50 mL beaker • Measuring tube (serological pipette) with a balloon (or pipettor) • Analytical balance (with the precision of the thousandth of gram) • Distilled (DI) water • Experimental liquid
Principle	• The drop of a mass (m) gets released when its weight G = mg is $\geq$ the surface force at the end of the tube. $m.g = 2\pi r\gamma$ (1) • γ can be calculated from the known water mass (m) and the radius of stalagmometer tube ® . • However, it was shown experimentally that only about 2/3 of the drop volume gets released. In addition, this portion is not constant for all the drops. On the other hand, the ratio of the m and γ is constant for all the liquids. So: $\frac{m_1}{\gamma_1} = \frac{m_2}{\gamma_2}$ (2) • Where m_1 and m_2 are the masses of drop of liquids 1 and 2 respectively, and γ_1 and γ_2 are the surface tensions corresponding to these liquids. • If the liquid with known γ is used for one of them (e.g. water□ γ□□□□), the γ of the experimental (unknown) liquid can be calculated from equation (3): $\gamma = \gamma_{H_2O} \times \frac{m}{m_{H_2O}}$ (3)
Procedures	1. Mount the clean and dry Stalagmometer on the vertical stand. 2. Weigh the mass of the beaker m_0. 3. Fill the beaker with DI water. Mount the tubing with balloon on the top end of stalagmometer.

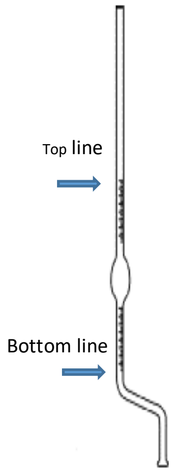
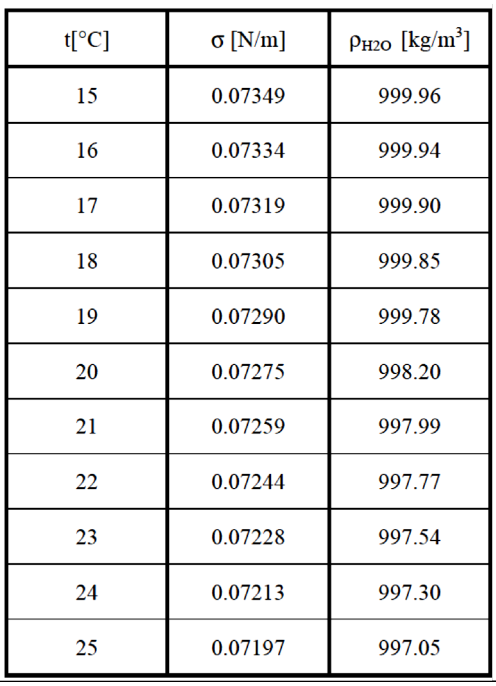
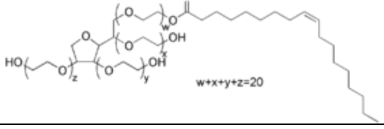
	4. Immerse the bottom end of stalagmometer into water and fill it up, such that the water level is above the wide part of stalagmometer. 5. Remove the balloon and collect 40 water drops into the beaker. 6. Weigh the mass of the beaker with water and determine the mass of 40 drops. 7. Empty the beaker and stalagmometer, dry them and prepare for the next measurement. 8. Repeat steps 2-6 for the experimental liquids with the unknown surface tension. 9. Knowing the lab temperature, determine the m_{H_2O} using values from the Table 1, and calculate the γ of unknown liquids according to the equation (3).
Notes	1. Use clean and dry glassware (contamination can wrong measurement) 2. Increasing # of drops will increase the precision of the measurements 3. Temperature can affect the surface tension values
Appendix	**Figure 1: Graduated Stalagmometer**  Stalagmometer with a calibrating

Table. 1: The temperature dependence of the surface tension and distilled water density



t[°C]	σ [N/m]	ρ_{H2O} [kg/m^3]
15	0.07349	999.96
16	0.07334	999.94
17	0.07319	999.90
18	0.07305	999.85
19	0.07290	999.78
20	0.07275	998.20
21	0.07259	997.99
22	0.07244	997.77
23	0.07228	997.54
24	0.07213	997.30
25	0.07197	997.05

LAB # 2

PPT 241: Physical Pharmacy Lab # 2 The Determination of the Surface Tension by Drop-Number Method	
Purpose	To measure the surface tension (γ) of Polysorbate 80 (Tween 80) solution using Stalagmometer  Chemical formula $C_{64}H_{124}O_{26}$ Molar mass 1310 g/mol
Requirements	- Stalagmometer (Figure 1) - 50 mL beaker - Measuring tube (serological pipette) with a balloon (or pipettor) - Distilled (DI) water - Experimental liquid
Principle	Known volume of the liquid (between 2 marks) is allowed to fall as drops from the tip of the stalagmometer under the effect of gravity and the number of drops is counted. For simplicity ρ1 and ρ2 can be disregarded if the reference liquid have density value close to those of the liquid under examination, therefore; Where: n2 = Number of drops of the unknown liquid n1 = Number of drops of the water γ1 = Surface tension of the water γ2 = Surface tension of the unknown liquid $\frac{\gamma_1}{\gamma_2} = \frac{n_2}{n_1}$
Procedures	1. Wash the stalagmometer several times with water before use. 2. Apply a suction bulb to the upper tip of stalagmometer 3. Introduce the flat tip of the stalagmometer into a small beaker (100 ml) half filled with distilled water, suck water through the apparatus (avoiding entrance of air bubbles) till level of water is 1 cm above mark of the apparatus. 4. Using filter paper, remove any humidity on the outside of the apparatus. 5. Adjust level of water to the upper mark and remove any suspended drop.

6. Count the number of water drops falling until the level of water reaches the lower mark (the suspended drop is not counted).
7. Repeat the procedure 3 times for water and calculate the mean number of drops.
8. Repeat the above steps for liquid A and B after washing the apparatus twice with the liquid to be measured.
9. Record the results in the table below and use equation (2) to calculate the surface tension of liquid A and B.
10. Represent the results graphically

Notes

4. Use clean and dry glassware (contamination can wrong measurement)
5. Increasing # of drops will increase the precision of the measurements
6. Temperature can affect the surface tension values

Appendix

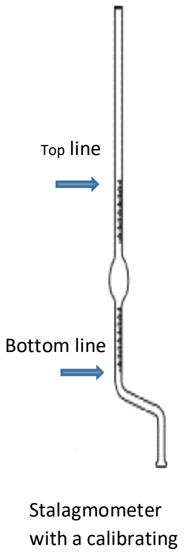
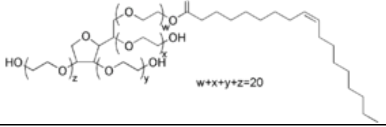


Figure 2: Graduated Stalagmometer

Results

Liquid	No. of drops				γ (dyne/cm)
	n_1	n_2	n_3	n_{mean}	
Water					72.8
Solution (1)					
Solution (2)					

LAB # 3

Ppt 241: Physical Pharmacy Lab # 3 The Determination of the Critical Micellar Concentration (CMC) Of Surfactant solution	
Purpose	To measure the Critical Critical Micellar Concentration (CMC)of Polysorbate 80 (Tween 80) solution using Stalagmometer (Drop Number Method)  Chemical formula $C_{64}H_{124}O_{26}$ Molar mass 1310 g/mol
Requirements	• Stalagmometer (Figure 1) • 50 mL beaker • Measuring tube (serological pipette) with a balloon (or pipettor) • Distilled (DI) water • Experimental liquid
Principle	• Surface active agents are molecules and ions that are adsorbed at interfaces, lowering the surface tension and decrease the contact angle between two phases. • Above a certain concentration range; surfactant molecules cannot be accommodated with ease, they begin to agglomerate in the bulk of the solution forming aggregates called micelle. • The lowest concentration at which micelles first appear is called the critical micelle concentration (CMC).

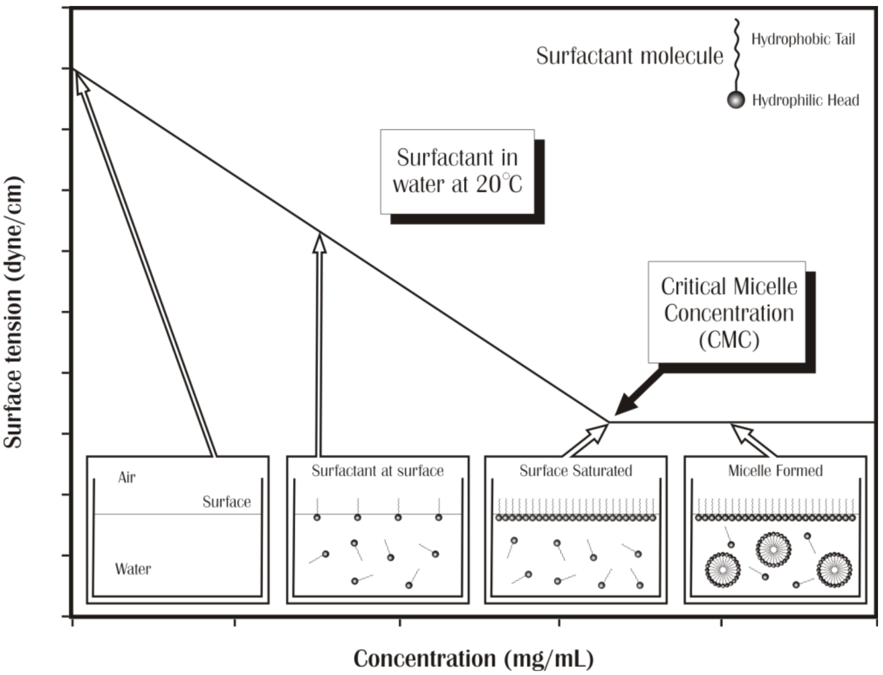


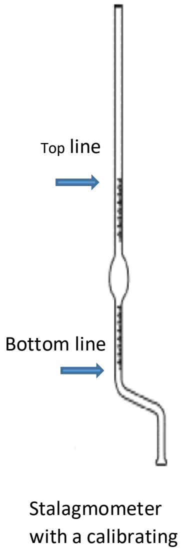
Figure1: Surface tension curve as a function of surfactant concentration.

Initially the surfactant molecules increasingly arrange themselves at the water surface. During this phase the surface tension decreases linearly with the increase in surfactant concentration. When the CMC is reached, i.e. when the surface is saturated with surfactant molecules, a further increase in surfactant concentration no longer has any appreciable influence on the surface tension.

The CMC can be determined by the surface tension method:

1. When the surfactant concentration is plotted against its respective surface tension, the surface tension decrease with increasing surfactant concentration until the CMC is reached after which the surface tension is nearly constant.
2. The determination is based on the method of drop weight and drop volume method:

$$\frac{\gamma_1}{\gamma_2} = \frac{n_2}{n_1}$$

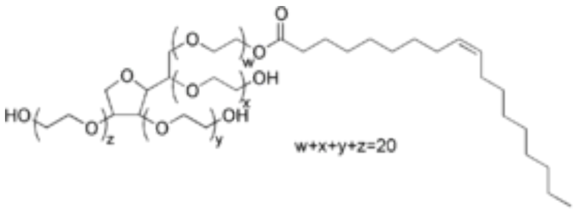
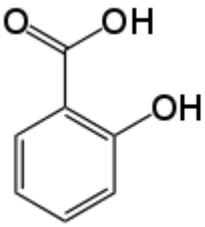
Procedures	- You are provided with different concentrations of a surfactant A. Determine CMC as follows: 1. Wash the stalagmometer several times with water before use. 2. Apply a suction bulb to the upper tip of stalagmometer 3. Introduce the flat tip of the stalagmometer into a small beaker (100 ml capacity) half-filled with distilled water. Suck the water through the apparatus (avoiding entrance of air bubbles) till the level of water become above the upper mark of the apparatus by about 1cm. 4. Using a small filter paper remove any attached humidity of the outside the stalagmometer, adjust the level and remove any suspended drop. 5. Count the number of drops falling until the level of water reaches the lower mark (N_1). 6. Repeat the procedure for the different concentratios and determined the number of drops (N_2) after washing stalagmometer with water. 7. Tabulate your result and calculate the average surface tension for the given surfactant concentrations provided that the surface tension of water = 72.8 dyne/ cm. 8. Draw the curve between the surface tension on Y axis and the concentration of surfactant on X axis and determine CMC
Notes	7. Use clean and dry glassware (contamination can wrong measurement) 8. Increasing # of drops will increase the precision of the measurements 9. Temperature can affect the surface tension values
Appendix	 Top line Bottom line Stalagmometer with a calibrating **Figure 2: Graduated Stalagmometer**

Results

Liquid	Conc.	No. of drops				γ (dyne/cm)
		n_1	n_2	n_3	n_{mean}	
Water						**72.8**
Solution (1)						
Solution (2)						
Solution (3)						
Solution (4)						
Solution (5)						

LAB # 4

Ppt 241: Physical Pharmacy Lab # 4
Micellar Solubilization of Salicylic acid

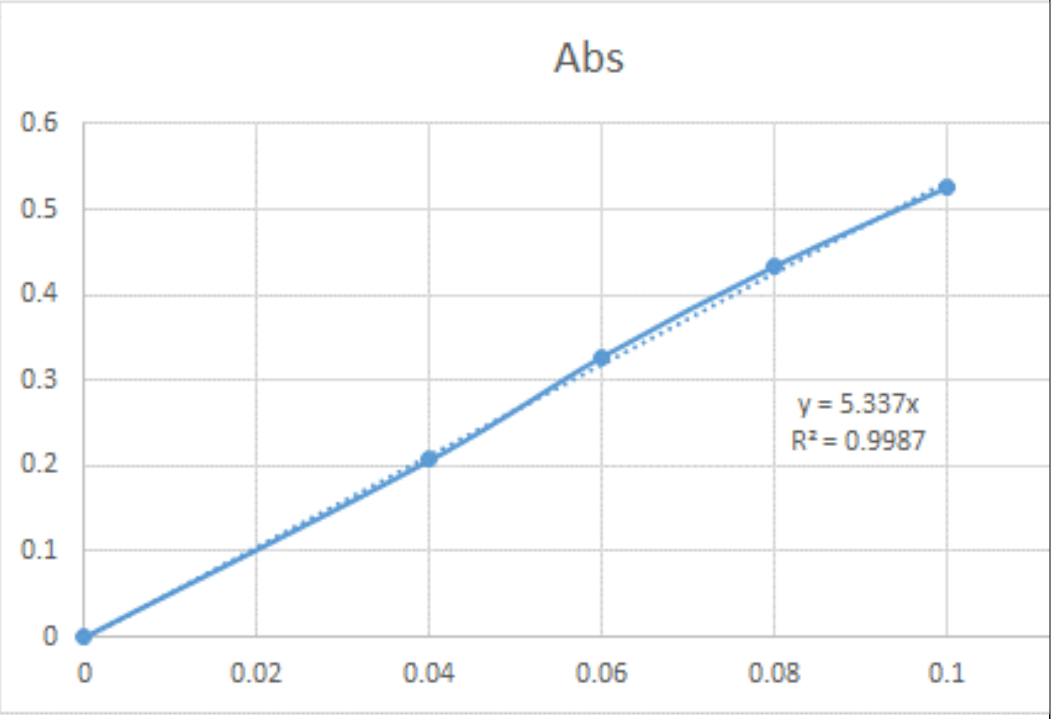
Purpose	• To measure the micellar solubilization capacity of Tween 80 in increasing the aqueous solubility of Salicylic acid • You recognize the effect of surface active agent (Tween 80) on the solubility of sparingly soluble drugs (Salicylic acid)  **Tween 80** Chemical formula $C_{64}H_{124}O_{26}$ Molar mass 1310 g/mol  **Salicylic acid** Chemical formula $C_{64}H_{124}O_{26}$ Molar mass 1310 g/mol
Requirements	• Salicylic acid • Tween 80 • 25m1 beaker • 6 (100 ml) beakers • Filter paper • Spectrophotometer with quartz cuvettes
Principle	• Water is a very powerful solvent. Water-surfactant solutions is used in the field of Pharmaceutics to make dosage forms containing sparingly soluble drugs. Generally, the degree of solubilization is a function of the physiochemical properties of the surfactant and solubilized drug. **Micelle Formation** • When the surface active agent molecules adsorbed as a monolayer in the water-air interface they become closely packed that additional molecules cannot be accommodated easily.

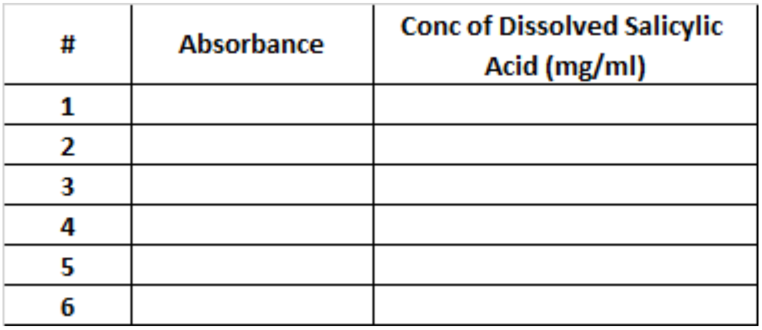
	• Polar groups pull the hydrocarbon chains partly into water. At certain concentration the interface and the bulk phase become saturated with monomers. Excess surface active agent agglomerates in solution forming aggregates (Micelle) & the free energy of the system is reduced. The lowest concentration at which micelles first appear is the critical Micelle concentration [CMC]. • At a given concentration, temperature, and salt content, all micelles of a given surfactant usually contain the same number of molecules, i.e. they are usually monodispersed. For different surfactants in dilute aqueous solutions, this number ranges approximately from 25 to 100 molecules. **Solubilization of Salicylic Acid by Tween 80:** • One of the properties of surfactant is the ability to increase the solubility of a sparingly soluble drug through micelle formation. Below CMC, as the SAA concentration increases the solubility of the compound increases slowly. At ≥ CMC a sharp increase in solubility can be observed due to the micelle formation.
Experimental	Work in group of 4, then share data with the group. 1. Number 6 clean 25 m1 beakers (1-6). 2. Place 250mg of salicylic acid in each beaker. 3. To each beaker add 25 ml of SLS (0%, 0.05, 0.1%, 0.2%, 0.3%, and 0.4%). 4. Shake well for 10 min. this will produce 1% salicylic acid (if the total amount dissolved). 5. Filter the solution of each beaker. 6. Take 1 ml of each filtrate and dilute with DI water to 100 ml. 7. Measure the absorbance of each filtrate solution.

Results

A. Standard Curve

- You will be given the UV absorbance's of salicylic acid standards, at 310 nm.
- Use water as a blank. Construct a Beer's Law plot of absorbance vs. concentration. Determine the slope, intercept, and R^2value.
- Determine the concentration of salicylic acid (SA) dissolved in each sample using values from your Beer's Law plot, and the following equation rearrangement:



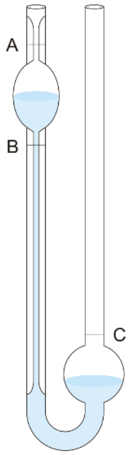


#	Absorbance	Conc of Dissolved Salicylic Acid (mg/ml)
1		
2		
3		
4		
5		
6		

$$\text{concentration} = \frac{\text{absorbance}}{\text{slope}}$$

Note

LAB # 5

PPT 241: Physical Pharmacy Lab # 5 **Determine the relative viscosity of pure liquids and solutions**	
Purpose	• To determine the Molecular Weight of a water soluble Polymer by using Capillary Viscometer (Ostwald viscometer)
Requirements	• Distilled water, samples (Different Water-soluble polymer solutions) , temperature-controlled 30°C water bath. Apparatus to hold viscometer vertically in water bath, thermometer, Capillary viscometer tube (Ostwald viscometer), Solvent wash bottle, Stopwatch (±0.2 sec).
Principle	Capillary Viscometer The viscosity of a Newtonian liquid may be determined by measuring the time required for the liquid to pass between two marks as it flows by gravity through a vertical capillary tube, known as an Ostwald viscometer The time of flow of the liquid under test is compared with the time required for a liquid of known viscosity (usually water) to pass between the two marks If η_1 nd η_2 are the viscosities of the unknown and the standard liquids, and t_1, and t_2 are the respective flow times in seconds, the viscosity $\eta_1$of the unknown liquid is called the relative η, is determined by substituting the experimental values in the equation $\frac{\eta_1}{\eta_2} = \frac{t_1}{t_2}$ 

Experimental

Work in group of 2, then share data with the group.
The sample is loaded into Ostwald viscometer. The time required for the sample to flow between two points on the viscometer is used to calculate the relative, viscosity.

1. Apply the suction bulb to tube B make suction to draw the sample through the capillary to a level of the start mark
2. Remove the suction bulb and measure the efflux time from starting mark (A to B) to the stop mark (in seconds).
3. The above procedure is done for both distilled water and the unknown samples each for three times and the mean of the three results is calculated
4. Put your results in table and calculate the relative viscosity in centipoises for each sample.

Results

Liquid	the time of flow				**η** (cps)
	t_1	**t_2**	**t_3**	**t_{mean}**	
Water					**1**(cps)
Liquid A (………..)					
Liquid B (………)					
Liquid C (………)					

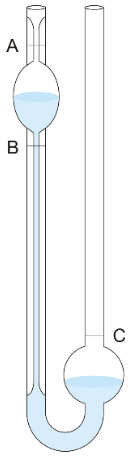
Calculate the relative viscosity in centipoises using the following simple relation

where
η_1 = viscosity of unknown sample
η_2 = viscosity of water
t_1 = the time in seconds of flow of unknown sample
t_2 = the time in seconds of flow of water

Note

Density values of samples
75% glycerin=(1.19g/cm³),
50% glycerin =(1.07g/cm³),
25% glycerin = =(1.05g/cm³),

LAB # 6

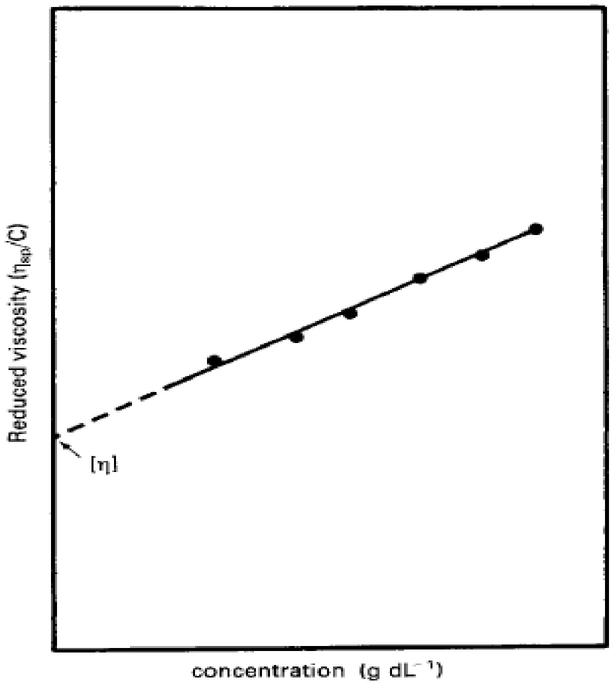
PPT 241: Physical Pharmacy Lab # 5 **Determination of Molecular Weight of a Polymer by Viscometery** **(Viscosity average molecular weight)**	
Purpose	• To determine the Molecular Weight of a water soluble Polymer by using Capillary Viscometer (Ostwald viscometer)
Requirements	• Distilled water, samples (Different Water-soluble polymer solutions) , temperature-controlled 30°C water bath. Apparatus to hold viscometer vertically in water bath, thermometer, Capillary viscometer tube (Ostwald viscometer), Solvent wash bottle, Stopwatch (±0.2 sec).
Principle	• When a polymer dissolves in a solvent, the viscosity of the solvent increases and polymer chain has direct influence. • **Hermanan Studinger** reported a relationship between polymer M.wt and the relative magnitude of increase of viscosity • If the bulk volume of the viscometer is fixed, (t and t_0) are the flow rates and (η and η_0) are the viscosities of polymer solution and pure solvent: The ratio $t / t_0 = \eta / \eta_0$ • Since the lower limit of η_{rel} is unity, the specific viscosity η_{sp} which defines the relative increase in viscosity brought about by dissolved polymer molecules $\eta_{sp} = \eta_{rel} - \eta_0 / \eta 0 = (\eta_{rel} - 1)$ • **The reduced viscosity** (η_{red}) is obtained by relating (η_{sp}) to concentration C. • $\eta_{red} = \eta sp/C$ 

Experimental

- Using water as a solvent to prepare different concentrations of a hydrophilic polymer A as seen in the table below (Use steps as in Lab 4)
- Calculate $\eta_{rel.}$, η_{sp} .and $\eta_{red.}$ For each solution and put the results in table
- Using a regular graph paper drew the (η_{red}) values for the different polymer concentrations on Y axis against conc (C). on X axis.
- Extrapolate the produced line to intersect with the Y axis to get the value of (η_{red}) at zero concentration which is the intrinsic viscosity.
- Apply in the Mark-Houwink equation to get the moleculer weight
- From this equation the molecular weight of a polymer can be determined from data on the intrinsic viscosity and vice versa.
- $\boldsymbol{\eta_{int.} = K.M^{a}}$
- where : η_{int} is the volume of polymer molecule in a given solvent
- M is the viscosity average molecular weight
- (K and a) are constants which describe the influence of solvent on the viscosity average molecular weight
- Values of (a) factor of Mark-Houwink equation are typically between 0.5 and 0.8
- Plot of concentration (g dL-1) against reduced viscosity η_{red} which by extrapolation gives the intrinsic viscosity η_{int}
- For the polymer used $a= 1$ and $K=10^3$
- $\eta_{int} =$ from figure
- M=??

Results

Conc. Of polymer solution in water solvent (C)	η_{rel}. $t / t_0 = \eta_{rel} / \eta_0$ $\eta_0 = 1$	η_{sp}. $\eta_{sp} = (\eta_{rel} — 1)$	η_{red}. $\eta_{red} = \eta_{sp}/C$
0.001 gm/dL			
0.002 gm/dL			
0.003 gm/dL			
0.004 gm/dL			
0.005 gm/dL			



Note

PPT-241 Pharmaceutics – I

(Physical Pharmacy)

Lab Report

Student Name: …………………………………………………………………… **Student #: ……………………………**

I did the lab work with: ………………………………………………….

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Lab #		**Date : ……../……../………………………**
Title:	…………………………………………………………………………………………………… …………………………………………………………………………………………………… …………………………………………………………………………………………………… ……………………………………………………………………………………………………	
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Results:

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Conclusion	Based on the results above we can conclude that …………………………………………………………………………………………………… …………………………………………………………………………………………………… …………………………………………………………………………………………………… …………………………………………………………………………………………………… …………………………………………………………………………………………………… ……………………………………………………………………………………………………
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